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Synthesis and Structures of Scandium and Lutetium Benzyl Complexes

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Supporting Information

Table S-1. Crystal data and structure refinement for **1**.

Empirical formula	C ₃₃ H ₄₅ O ₃ Sc
Formula weight	534.67
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, C c
Unit cell dimensions	a = 17.202(4) Å alpha = 90 deg. b = 20.759(5) Å beta = 93.695(4) deg. c = 8.267(2) Å gamma = 90 deg.
Volume	2946.0(12) Å ³
Z, Calculated density	4, 1.205 Mg/m ³
Absorption coefficient	0.280 mm ⁻¹
F(000)	1152
Crystal size	0.53 x 0.16 x 0.10 mm
Theta range for data collection	2.84 to 27.10 deg.
Limiting indices	-22 ≤ h ≤ 21, -26 ≤ k ≤ 26, -10 ≤ l ≤ 10
Reflections collected / unique	12243 / 5994 [R(int) = 0.0387]
Completeness to theta = 25.00	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9724 and 0.8519
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	5994 / 2 / 514
Goodness-of-fit on F ²	1.009
Final R indices [I > 2sigma(I)]	R1 = 0.0399, wR2 = 0.0824
R indices (all data)	R1 = 0.0471, wR2 = 0.0852
Absolute structure parameter	0.03(2)
Largest diff. peak and hole	0.228 and -0.234 e.Å ⁻³

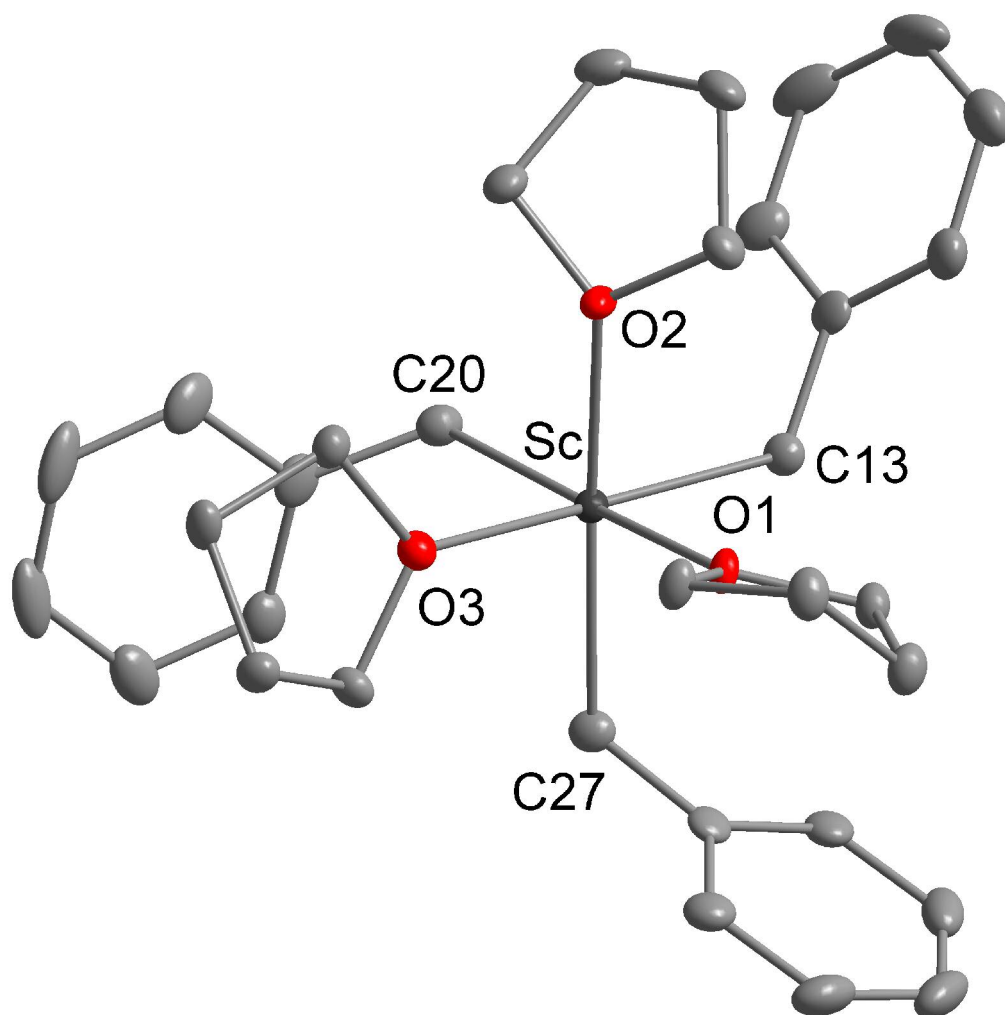


Figure S-1. Molecular structure of **1**, with 50% probability ellipsoids. Hydrogen atoms are omitted for clarity.

Table S-2. Crystal data and structure refinement for **2**.

Empirical formula	C ₂₉ H ₃₇ O ₂ Sc
Formula weight	462.57
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P $\bar{1}$
Unit cell dimensions deg. 82.664(1) deg. deg.	a = 7.6779(6) Å alpha = 86.423(1) b = 11.3723(9) Å beta = c = 14.824(1) Å gamma = 78.332(1)
Volume	1256.35(16) Å ³
Z, Calculated density	2, 1.223 Mg/m ³
Absorption coefficient	0.315 mm ⁻¹
F(000)	496
Crystal size	0.47 x 0.27 x 0.21 mm
Theta range for data collection	2.73 to 28.28 deg.
Limiting indices	-10 ≤ h ≤ 10, -15 ≤ k ≤ 14, -19 ≤ l ≤ 18
Reflections collected / unique	11481 / 5968 [R(int) = 0.0164]
Completeness to theta = 25.00	98.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9359 and 0.8622
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	5968 / 0 / 437
Goodness-of-fit on F ²	1.040
Final R indices [I > 2σ(I)]	R ₁ = 0.0418, wR ₂ = 0.1043
R indices (all data)	R ₁ = 0.0502, wR ₂ = 0.1105
Absolute structure parameter	?
Largest diff. peak and hole	0.732 and -0.458 e.Å ⁻³

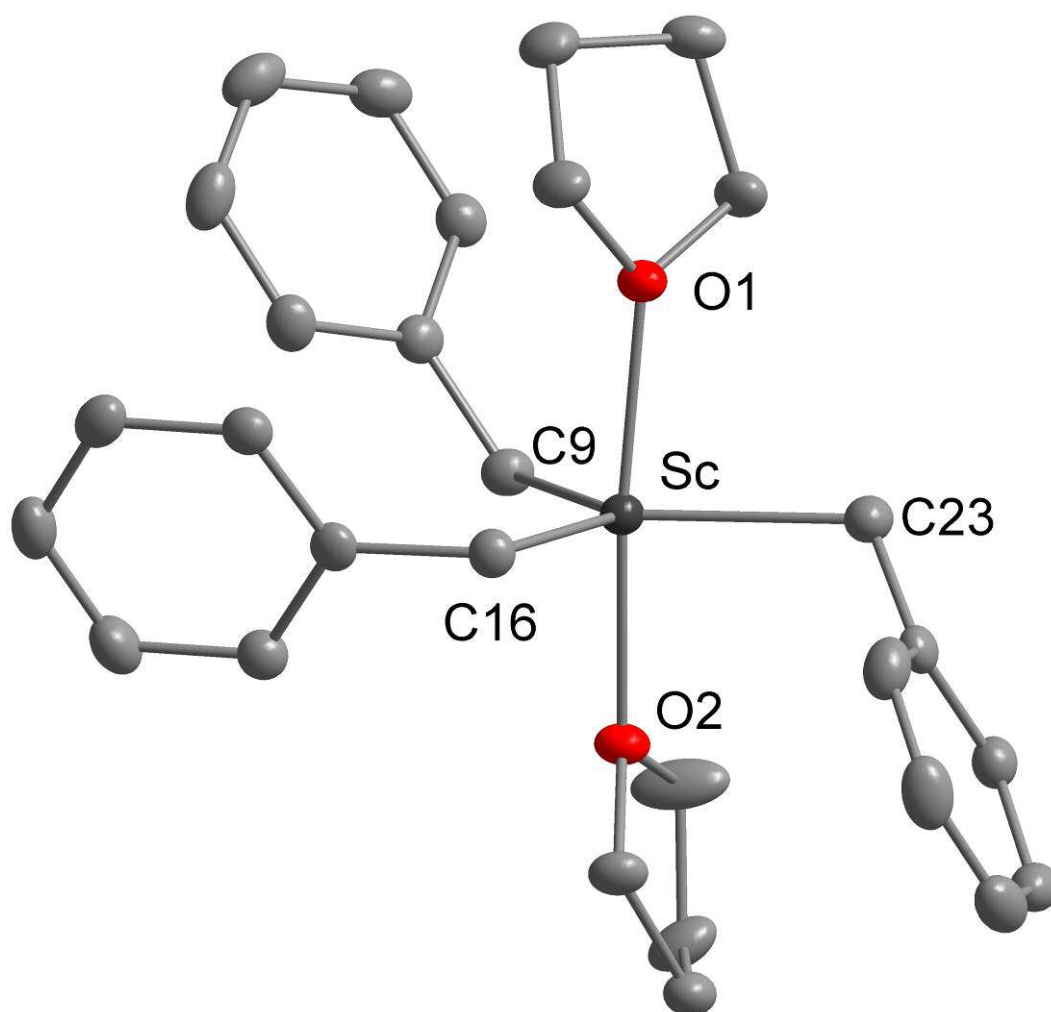


Figure S-2. Molecular structure of **1**, with 50% probability ellipsoids. Hydrogen atoms are omitted for clarity.

Table S-3. Crystal data and structure refinement for **3**.

Empirical formula	C ₄₇ H ₅₉ K ₂ O ₃ Sc
Formula weight	795.10
Temperature	200(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, Pna2(1)
Unit cell dimensions	a = 25.471(5) Å alpha = 90 deg. b = 9.2954(19) Å beta = 90 deg. c = 18.336(4) Å gamma = 90 deg.
Volume	4341.3(15) Å ³
Z, Calculated density	4, 1.216 Mg/m ³
Absorption coefficient	0.399 mm ⁻¹
F(000)	1696
Crystal size	0.5 x 0.15 x 0.1 mm
Theta range for data collection	2.93 to 29.25 deg.
Limiting indices	-28 ≤ h ≤ 34, -12 ≤ k ≤ 11, -21 ≤ l ≤ 25
Reflections collected / unique	19529 / 10588 [R(int) = 0.0493]
Completeness to theta = 29.25	98.4 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	10588 / 1 / 446
Goodness-of-fit on F ²	0.939
Final R indices [I > 2sigma(I)]	R ₁ = 0.0568, wR ₂ = 0.1269
R indices (all data)	R ₁ = 0.1011, wR ₂ = 0.1427
Absolute structure parameter	0.01(4)
Largest diff. peak and hole	1.002 and -0.433 e.Å ⁻³

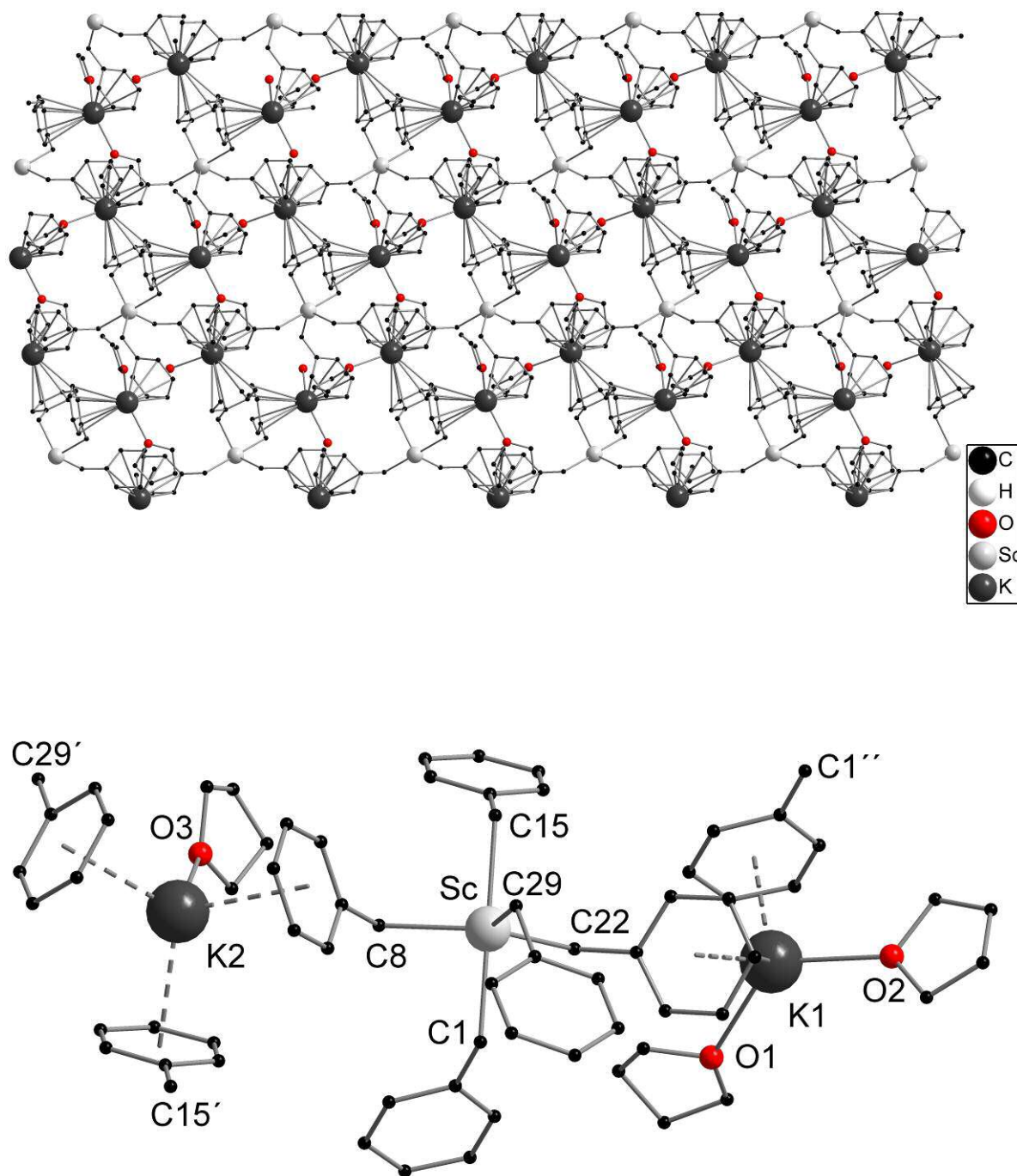


Figure S-3. Solid-state structure of **3**. Top: View to a single sheet from above. Bottom: Shown is one unit out of the polymeric structure and the atom labeling scheme, omitting hydrogen atoms.

Table S-4. Crystal data and structure refinement for **4**.

Identification code	z:\daten\nils\nils52b\nils52b
Empirical formula	C ₃₃ H ₄₅ Lu O ₃
Formula weight	664.66
Temperature	200(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, C c
Unit cell dimensions	a = 17.3716(9) Å alpha = 90 deg. b = 20.9550(14) Å beta = 93.571(4) deg.
Volume	3050.3(3) Å ³
Z, Calculated density	4, 1.447 Mg/m ³
Absorption coefficient	3.266 mm ⁻¹
F(000)	1352
Crystal size	0.458 x 0.240 x 0.079 mm
Theta range for data collection	2.81 to 25.02 deg.
Limiting indices	-20 ≤ h ≤ 20, -24 ≤ k ≤ 24, -9 ≤ l ≤ 9
Reflections collected / unique	13424 / 5033 [R(int) = 0.0381]
Completeness to theta = 25.02	99.5 %
Absorption correction	Integration
Max. and min. transmission	0.7403 and 0.4640
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	5033 / 2 / 334
Goodness-of-fit on F ²	0.985
Final R indices [I > 2sigma(I)]	R1 = 0.0203, wR2 = 0.0449
R indices (all data)	R1 = 0.0228, wR2 = 0.0454
Absolute structure parameter	-0.001(8)
Largest diff. peak and hole	0.395 and -0.669 e.Å ⁻³

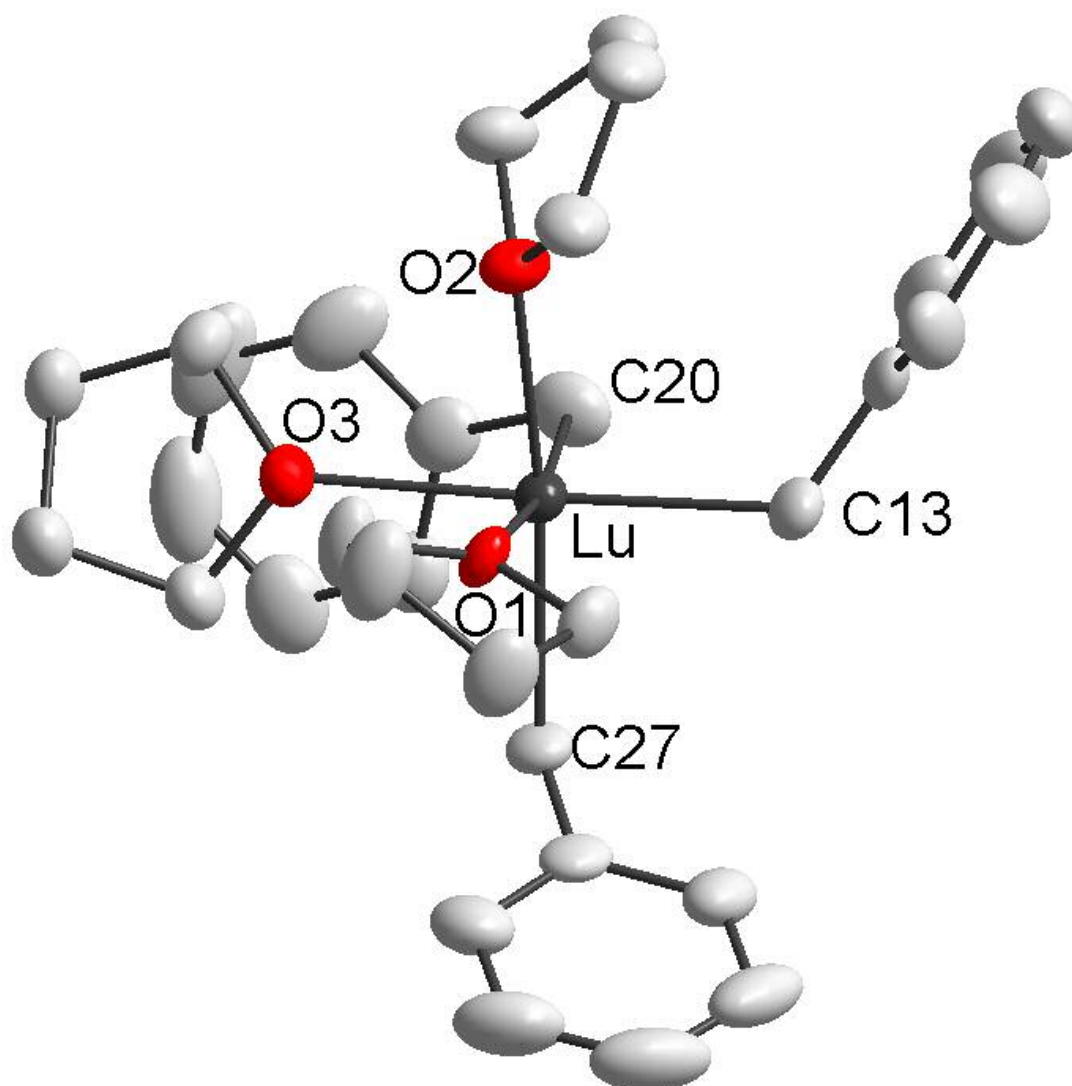


Figure S-4. Molecular structure of **4**, with 50% probability ellipsoids. Hydrogen atoms are omitted for clarity.

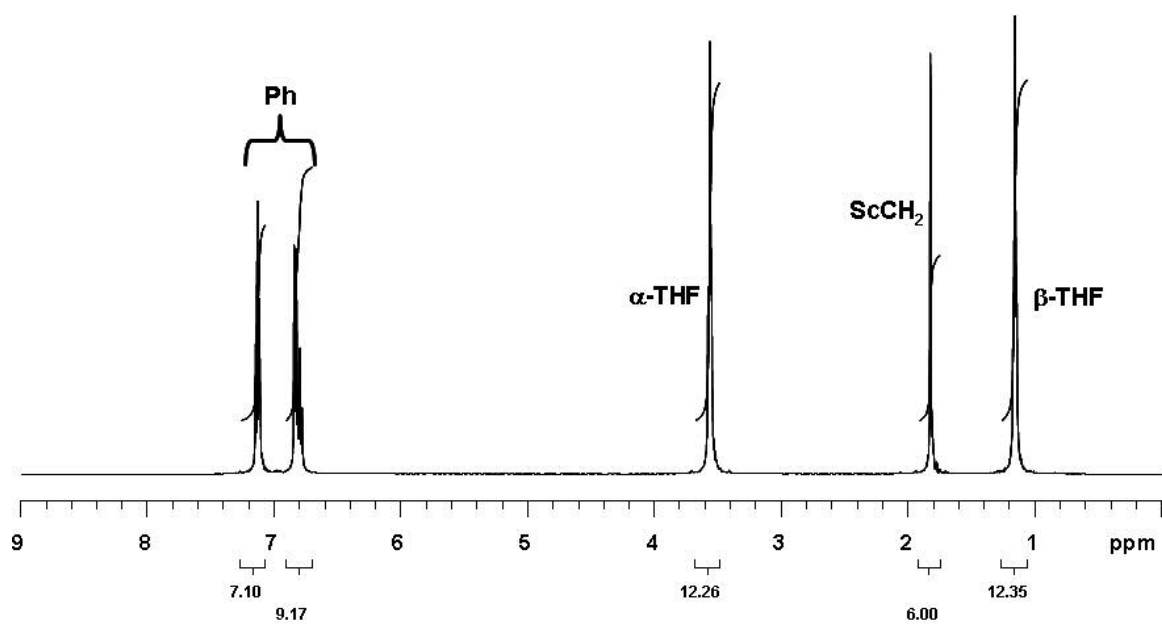


Figure S-5. ¹H NMR spectrum (500 MHz, C₆D₆, 20°C) of Sc(CH₂Ph)₃(THF)₃ (**1**).

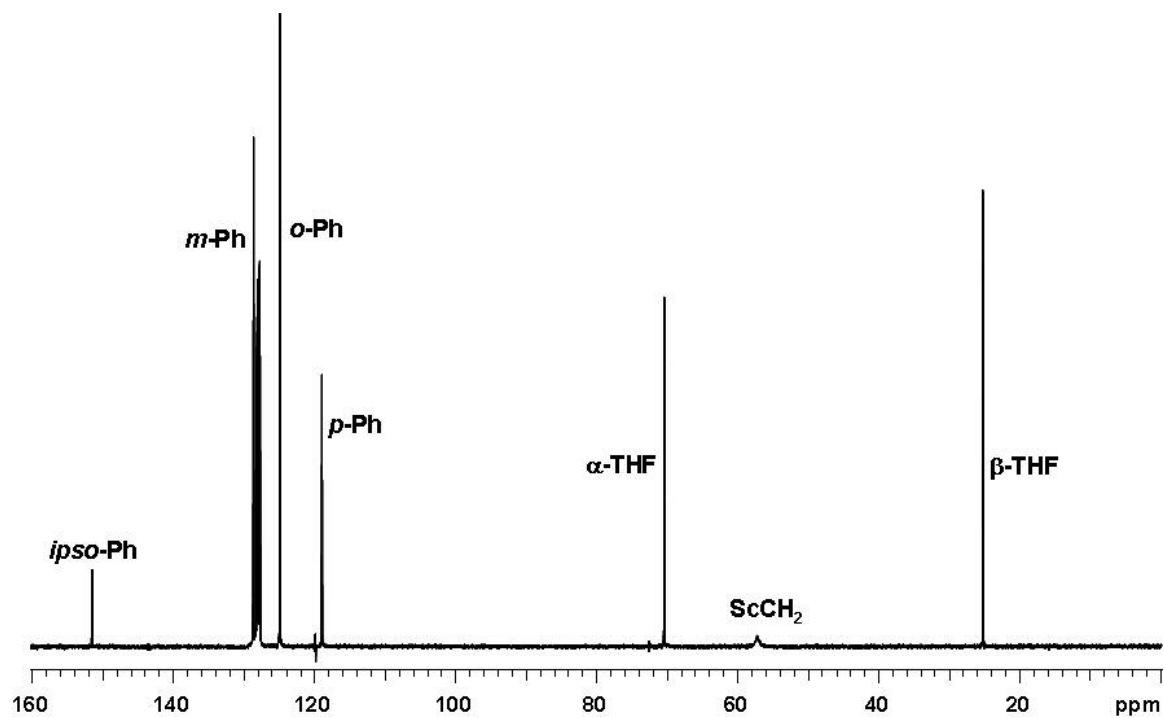


Figure S-6. ¹³C{¹H} NMR spectrum (125.7 MHz, C₆D₆, 20°C) of Sc(CH₂Ph)₃(THF)₃ (**1**).

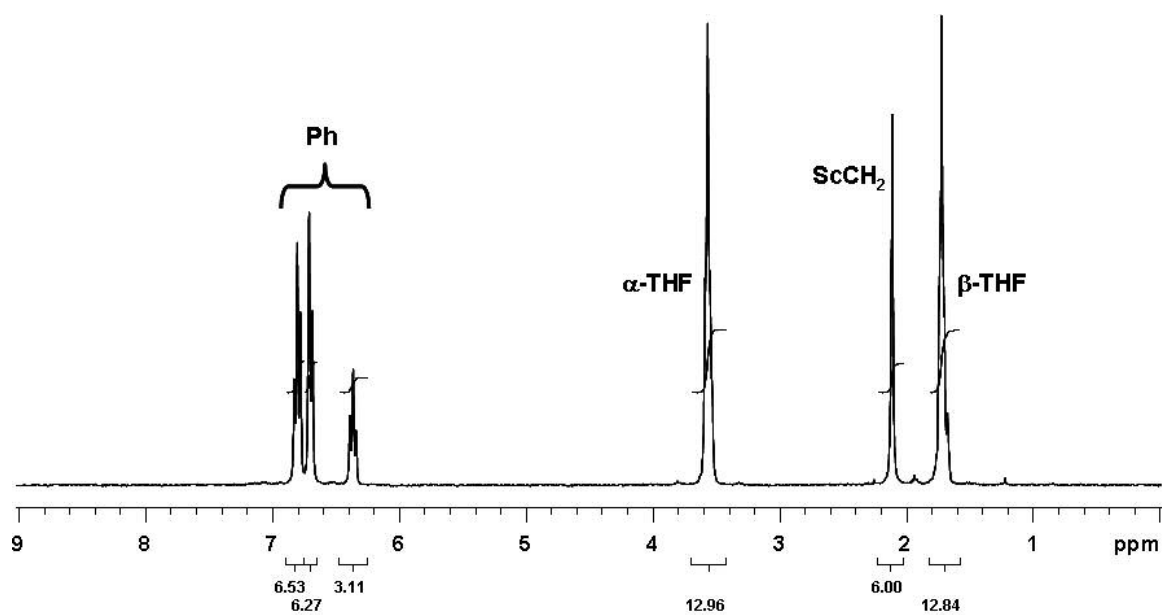


Figure S-7. ¹H NMR spectrum (500 MHz, THF-*d*₈, 20°C) of Sc(CH₂Ph)₃(THF)₃ (**1**).

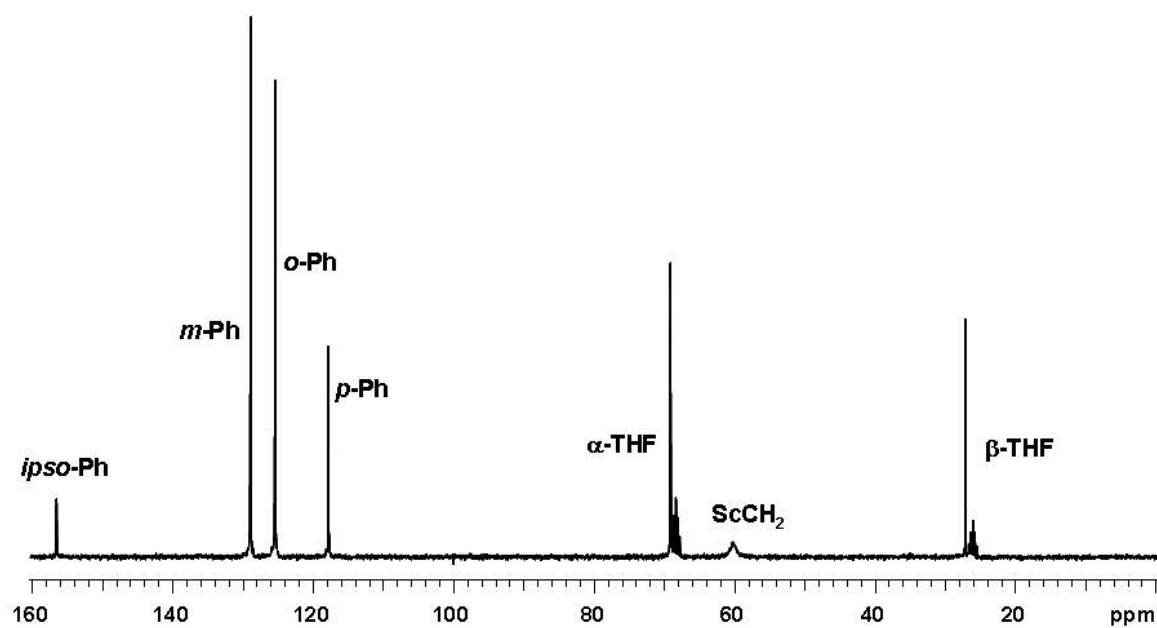


Figure S-8. ¹³C{¹H} NMR spectrum (125.7 MHz, THF-*d*₈, 20°C) of Sc(CH₂Ph)₃(THF)₃ (**1**).

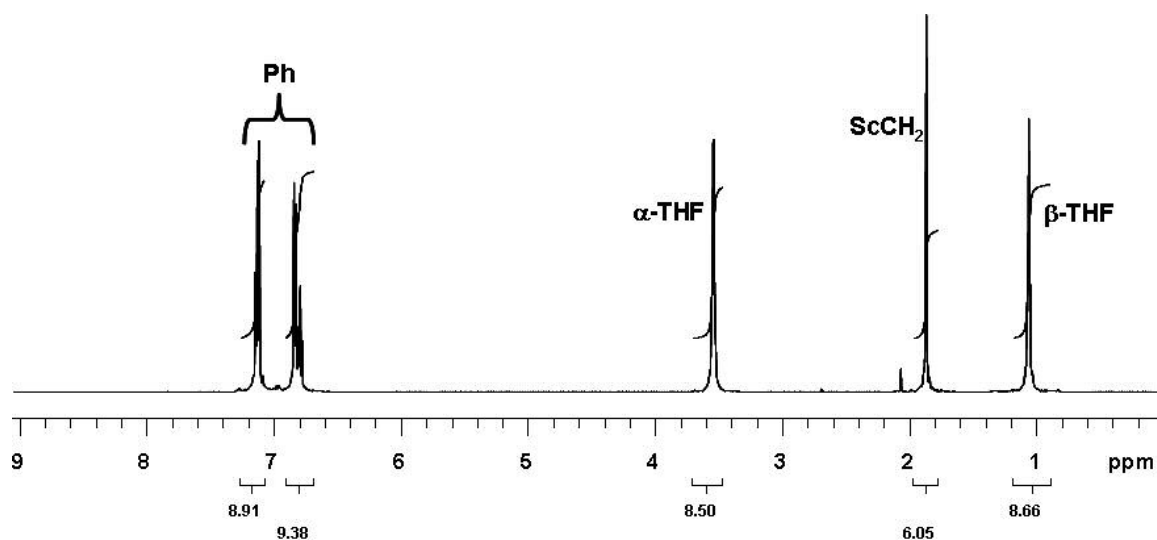


Figure S-9. ¹H NMR spectrum (500 MHz, C₆D₆, 20°C) of Sc(CH₂Ph)₃(THF)₂ (**2**).

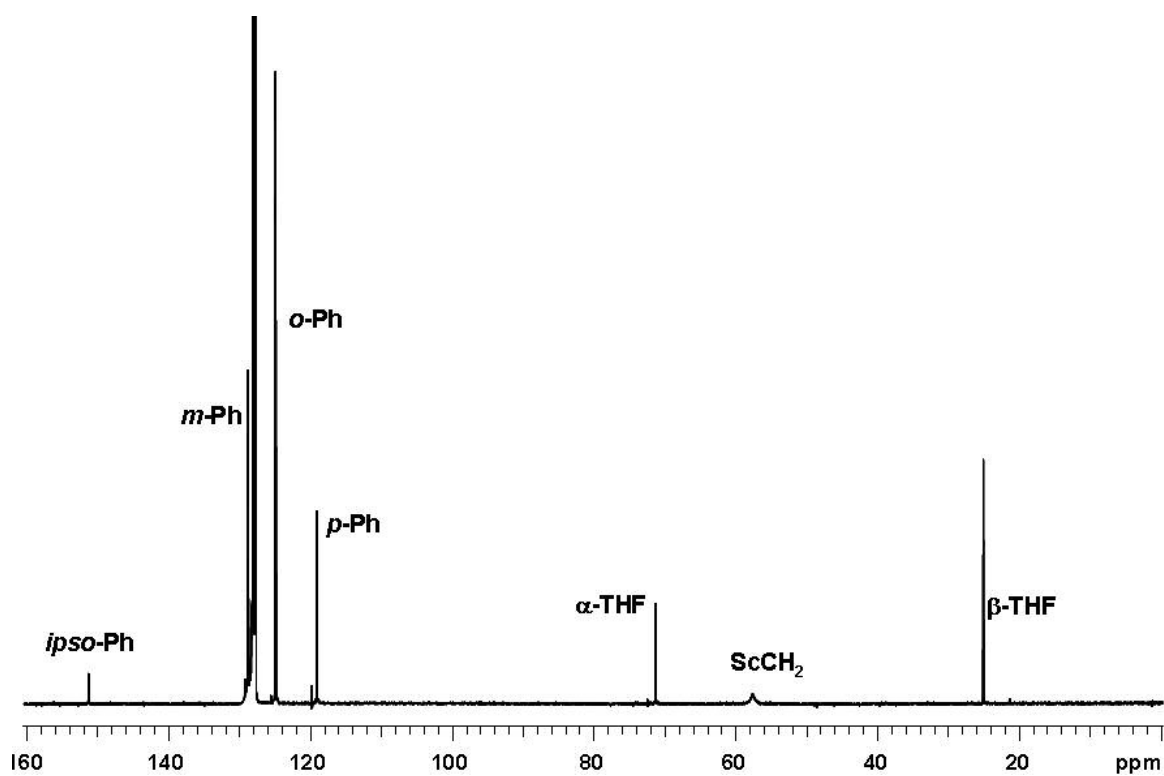


Figure S-10. ¹³C{H} NMR spectrum (125.7 MHz, C₆D₆, 20°C) of Sc(CH₂Ph)₃(THF)₂ (**2**).

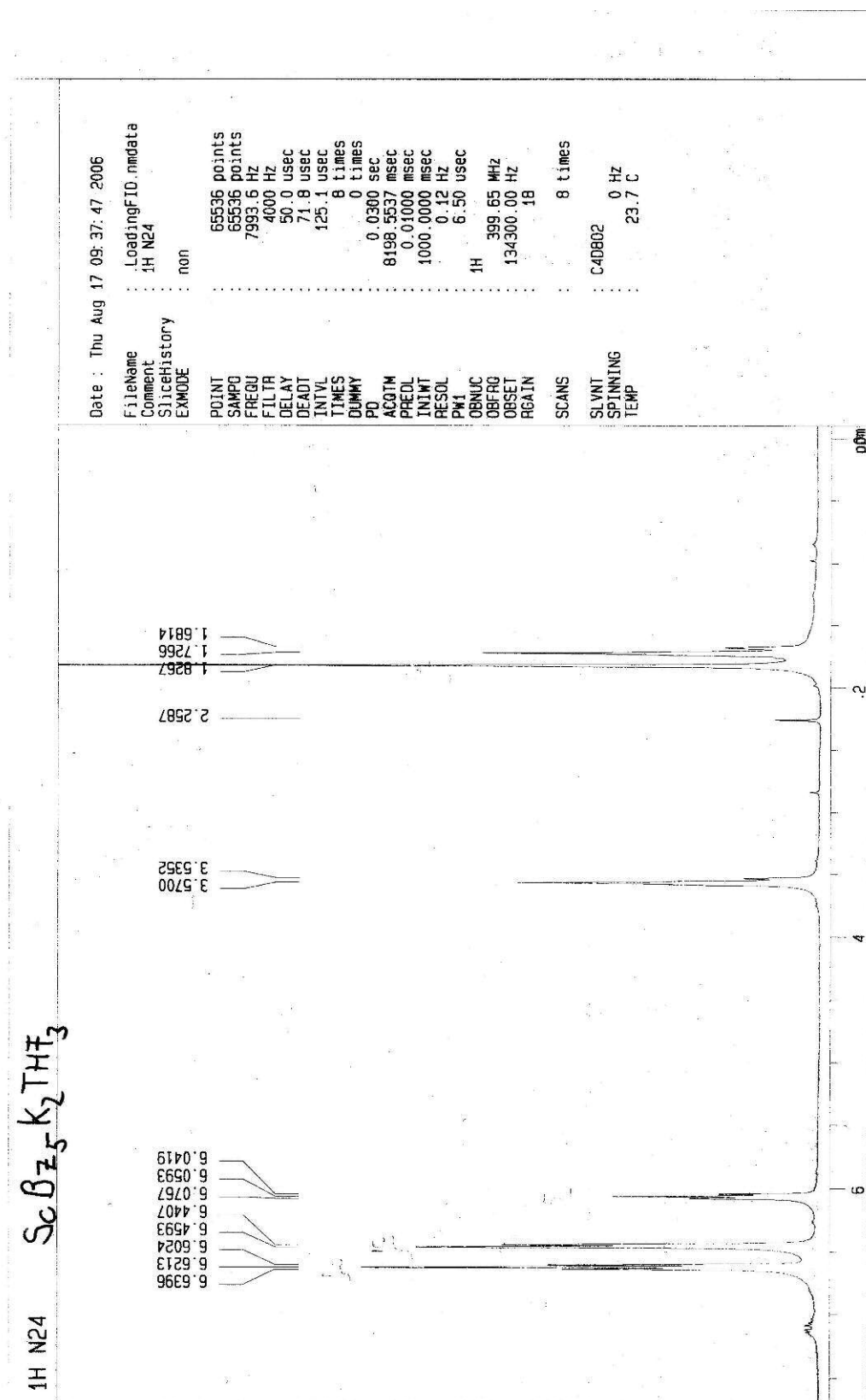
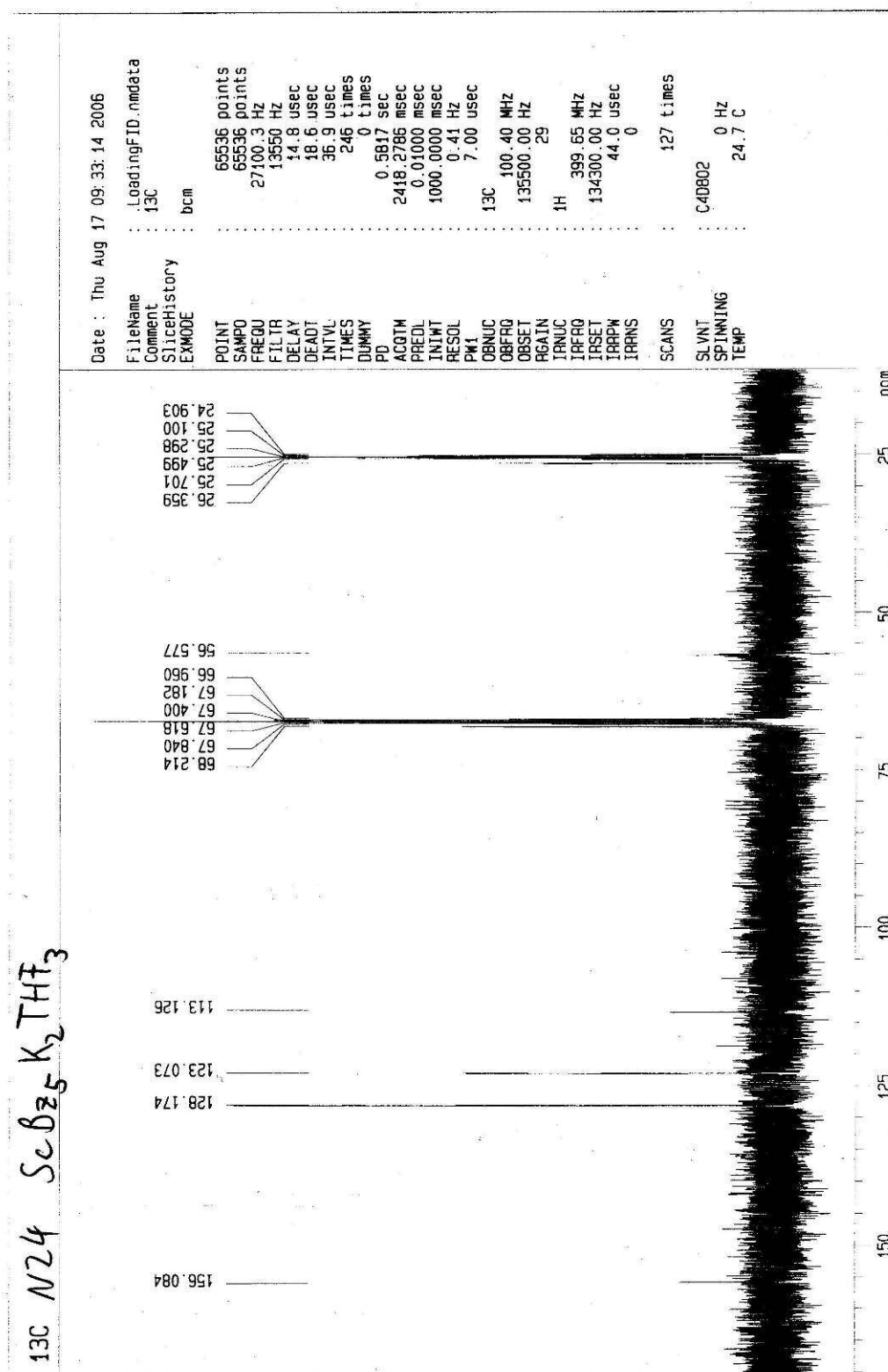
Figure S-11. ^1H NMR spectrum of **3**

Figure S-12. ^{13}C NMR spectrum of **3**

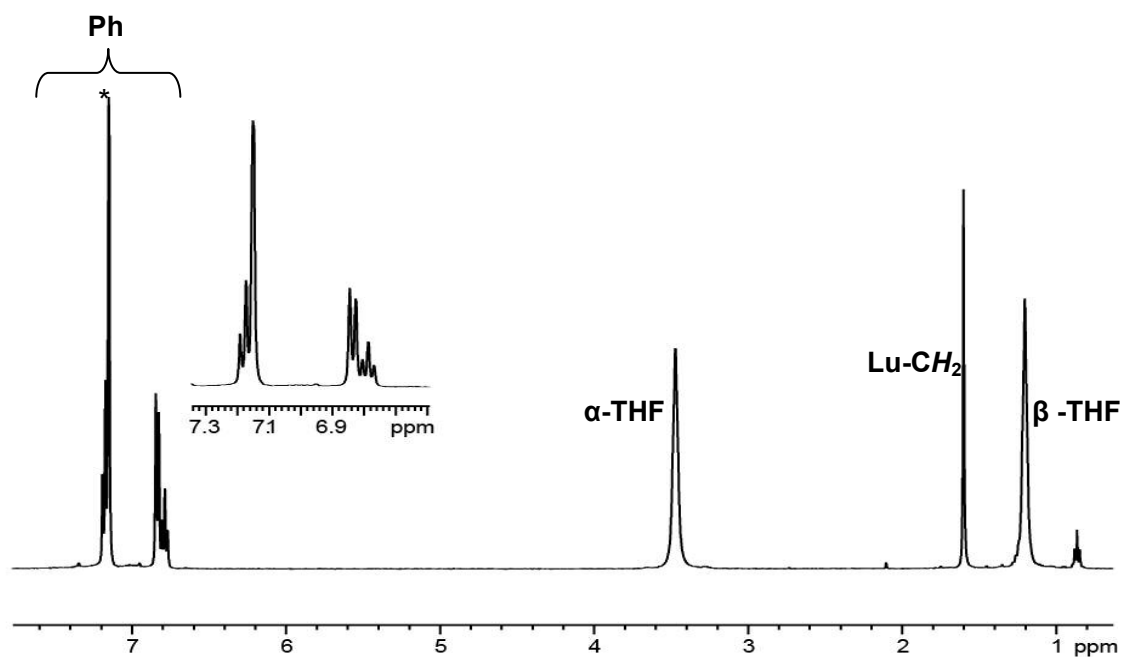


Figure S-13. ^1H NMR spectrum (400 MHz, C_6D_6 , 27°C) of $\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_3$ (4).

* denotes residual solvent signal.

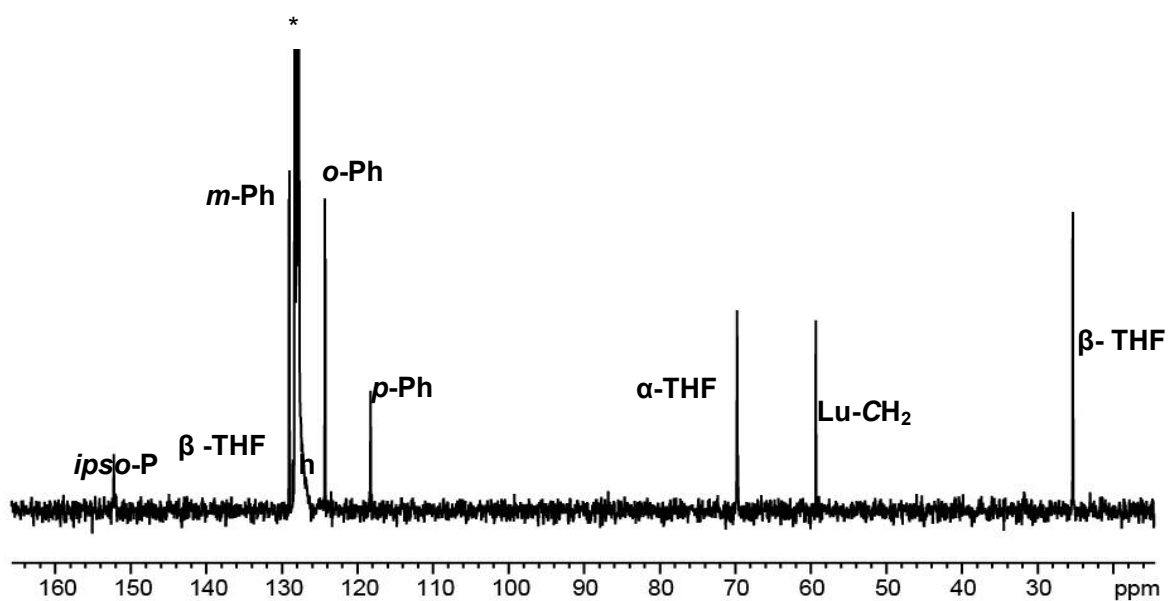


Figure S-14. ^{13}C NMR spectrum (100.58 MHz, C_6D_6 , 27°C) of $\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_3$ (4).

* denotes residual solvent signal.

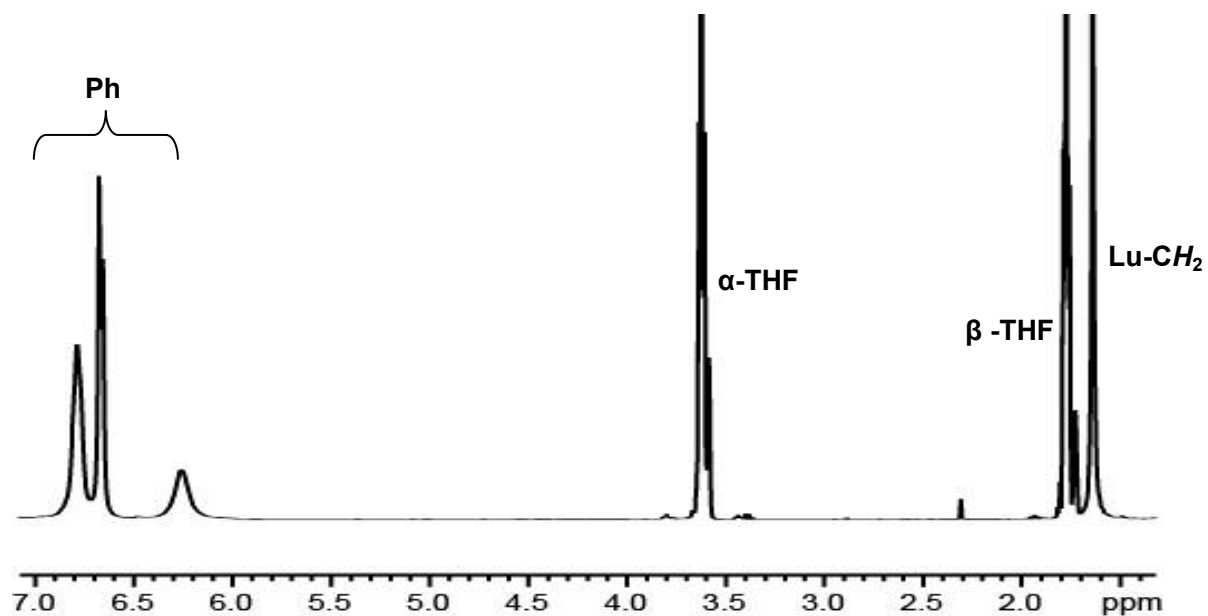


Figure S-15. ^1H NMR spectrum (400 MHz, $\text{THF}-d_8$, 27°C) of $\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_3$ (4).

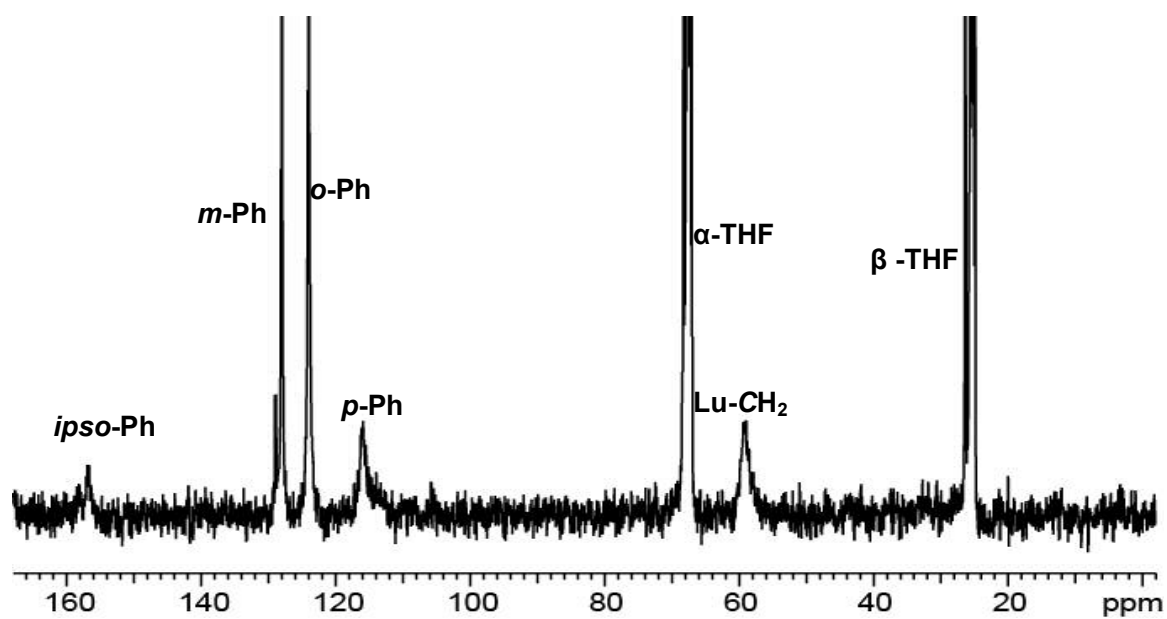


Figure S-16. ^{13}C NMR spectrum (100.58 MHz, $\text{THF}-d_8$, 27°C) of $\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_3$ (4).

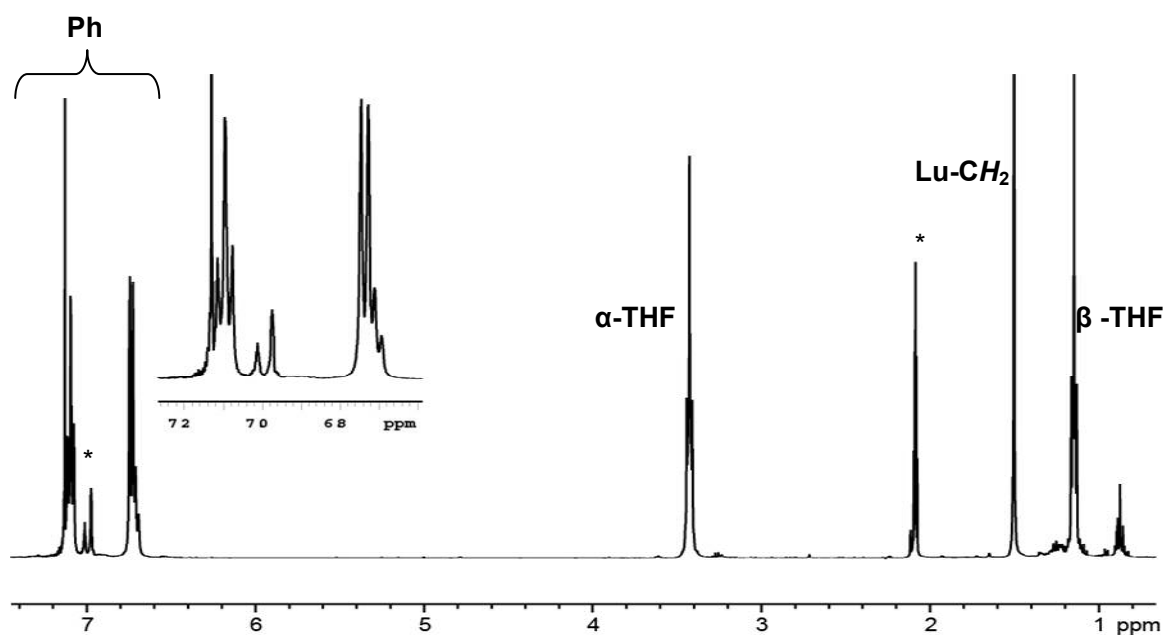


Figure S-17. ^1H NMR spectrum (400 MHz, Toluene- d_8 , 27°C) of $\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_2$ (5).
* denotes residual solvent signals.

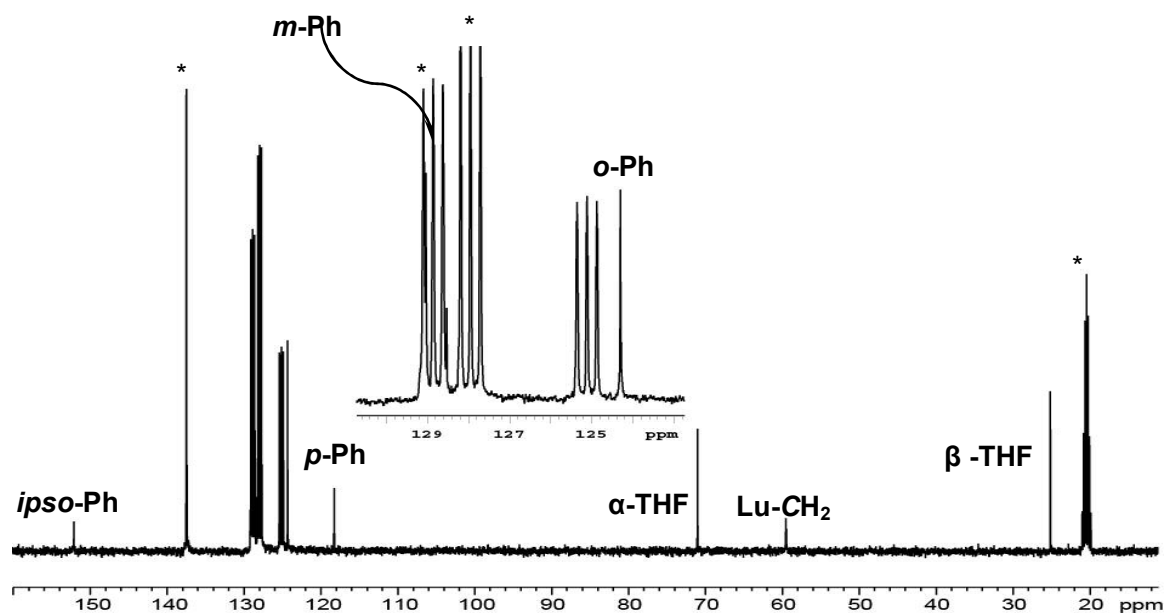


Figure S-18. ^{13}C NMR spectrum (100.58 MHz, Toluene- d_8 , 27°C) of $\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_2$ (5).
*denotes residual solvent signals.